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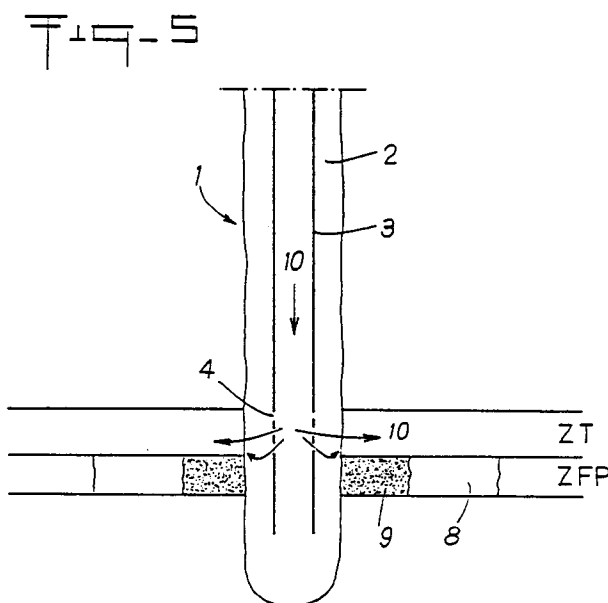
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F-42003 Saint-Etienne Cédex 1(FR)(54) **Product and process for acid diversion in the treatment of subterranean formations.**

(57) The invention relates to the treatment by a fluid, or more generally by an acid fluid, of subterranean formations located in particular around an oil, gas, water or geothermal well.

Acid is diverted towards the zone to be treated by a surfactant flush, the injection of a foam and

then the injection of an acid/surfactant composition. These operations are repeated as often as required, increasing at each stage representing approximately 80 cm (20 to 25 inches) of treatment in terms of height the quantity of foam injected.

Better diversion. Lower cost.

**EP 0 475 503 A1**

Product and process for acid diversion in the treatment of subterranean formations.

The present invention relates to the technical field of treatment of subterranean formations using a fluid, in particular an acid fluid.

These treatments are notably useful in the petroleum and associated industries. They are used to increase the production of the well and/or to restore the production of the well when this production declines in time down to an unacceptable level of return.

There are a number of causes for the decline in production.

Within the framework of the invention, two main categories can be contemplated : a reduction in the permeability of the oil "reservoir", or the invasion of this reservoir by the water contained in a lower layer.

A reduction in permeability is due, among other things, to the entrainment of fines, by the flow of the oil, towards the production well. Around this well, these particles accumulate and gradually plug the natural pores in the rock. The oil can then no longer flow out satisfactorily through this well. These particles can be of various origins (type of rock, damage to the reservoir, progressive deterioration of the rock, etc.)

Their origin matters little, the invention being directed to the treatment, using an acid fluid, of the rocks containing particles that are at least partially soluble in this fluid in order to cause these particles to dissolve totally or partially, and thus make the natural pores in the rock re-open to such an extent as to obtain an acceptable measure of permeability.

The invasion by water of the oil producing zone is due to the difference in viscosity between oil and water. The latter, which is far more mobile than oil, is progressively entrained towards the perforations in the well by the very flow of oil towards the well. This phenomenon is known as "water coning". It will be seen below that the invention also enables this problem to be overcome.

All the above is perfectly familiar to a man of the art, with regard both to the problems encountered and current solutions.

There is no need, either, to discuss in detail the structure of an oil well (or the like), as such a structure is also familiar to a man of the art.

The invention will be more readily understood from reading the following description and by referring to the annexed drawing, wherein :

- Figure 1 is a cross-sectional view of a well and of two subterranean formations crossed by this well.
- Figure 2 represents an acid treatment operation in which the acid treatment fluid (FT) pumped into the zone to be treated (ZT) is

prevented from penetrating a zone of higher permeability (SFP).

- Figure 3 represents a first stage (1) in which a surfactant "preflush" (8) is injected into zones ZFP and ZT.
- Figure 4 represents a second stage (2) in which a foam (9) is injected after the surfactant.
- Figure 5 represents a fourth stage (4) in which, as explained below, the foam (9) is capable of resisting, in zone ZFP, the injection of the acid fluid according to the invention (acid + surfactant). On the other hand, in zone ZT, the foam cannot withstand injection of the acid fluid (particularly if an intermediate well "shut-in" stage 3 has been observed).

The acid fluid (10) is thus diverted.

- Figure 6 represents the flow rate of acid fluid in zones ZT and ZPF, with shut-in (Figure 6a) and without shut-in (Figure 6b).- Without shut-in (Figure 6b), the diversion takes much longer to become established.
- Figure 7 represents the flow rate of acid fluid in zone ZT and zone ZFP when the acid fluid contains a surfactant (Figure 7b) or no surfactant (Figure 7a). It can be seen that, without a surfactant, the diversion is only ephemeral. This figure shows a diversion in the absence of oil (petroleum).
- Figure 8 (8a, 8b, 8c) represents an extension of the invention to the acid treatment of three superposed layers ZT1, ZT2 and ZT3.
- Figure 9 (9a, 9b) shows a diversion treatment according to the invention of a well partially invaded by water ("water coning").
- Figure 10 represents the results of the treatment shown in figure 9, simulated under laboratory conditions on the basis of the characteristics of a known well that had previously undergone treatment for "water coning" using a prior art method.
- Figure 11 represents a preferred form of embodiment of the invention.

In the figures, the same references designate the same elements throughout, these being as follows :

1. drilled well
2. cemented annulus
3. metallic casing
4. perforations through the casing and the cement placing the interior of the casing in communication with the producing zone
5. pattern of oil flow towards the well (5a) and towards the surface (5b)
- 6a. preferential flow of the treatment fluid FT in the zone (ZFP) the permeability of which is greater than that of the zone to be treated (ZT)

6b. flow of the treatment fluid (FT) towards the zone to be treated (ZT) thanks to the "diversion" caused by the plugging of the high permeability zone (ZFP) by the diversion agent (7)

7. diversion agent

FT. Treatment fluid

ZT. Zone to be treated

ZFP. Zone with permeability higher than that of the zone to be treated

ZT1, ZT2, ZT3 : three successive zones to be treated (Figure 8)

8. surfactant

9. foam

10. fluid acid

11. perforations

12. curve corresponding to the water cell

13. injection of surfactant

14. injection of foam

15 injection of treatment fluid + surfactant

16. curve corresponding to the oil cell

In the process under consideration, the action i) of a surfactant "preflush", ii) of a diversion agent formed by a foam and iii) of an acid treatment fluid itself comprising a certain proportion of surfactant, is combined.

Conventionally, one can also inject a preflush of mutual solvents as a preliminary stage.

It is also useful to introduce an acid pumping stage between the said preliminary stage and stage 1. This "acid stage" can conventionally contain the following : HCl ; or HCl followed by "mud acid" (HCl + HF) ; or HCl followed by "clay acid" (fluoroderivative(s) of boric acid, especially fluoroboric acid) ; and similar combinations such as HCl followed by mud acid and then clay acid. The injection proportions and volumes to be used are well known to a man of the art. As to clay acid (acid composition for stabilizing clays), reference will be made to USP 4 151 879; 4 151 878 and 4 160 483.

In the case of gas wells, the preliminary stage and the acid stage can contain up to 50-70 % of a lower alcohol, preferably methanol.

Naturally, the conventional additives (anti-corrosion agents, clay stabilizers, clay and fines control agents ; anti-paraffinic agents, etc.) can be incorporated in the aforementioned stage in the known way.

Similarly, according to the nature of the subterranean formations, the man of the art will know whether he is to intercalate preflush stages such as : preliminary cleaning fluid, NH_4Cl together with conventional additives, etc.

Preferably, the chosen stages will contain an addition of one surfactant at least, absolute preference being given to the surfactant used to form the foam. Injections of nitrogen can also be used in a known manner to "nitrify" the fluids used.

Stage 1 can also be combined with the acid stage to simplify the method. For example, HCl can be added to the stage 1 preflush, or again one could add HCl + mud acid (or mud acid for gas wells).

Naturally, the pumping in stage 4 (acid + surfactant) can be carried out - and this is, moreover often recommended - in several sequences, it being essential for each sequence to include an effective proportion of surfactant.

By way of a non-limitative example, stage 4 can comprise :

- (acid 1 + surfactant) followed by (acid 2 + surfactant)
- (acid 1 + surfactant) followed by (acid 2).

Acid 1 is generally HCl.

Acid 2 can be HF, HCl or fluoroboric acid.

These stages can be repeated.

The invention has two main applications.

1. Diversion, as represented in figure 2. The invention also enables several layers or zones the permeability and extent of deterioration of which differ to be treated selectively. The object is to inject into each layer the volume of acid treatment fluid that corresponds to the specific deterioration of the said layer.

This problem is difficult to solve as the fluid will have a natural tendency to invade, not the target layer, but the other layer or layers that are less damaged, hence easier to penetrate.

2. Well partially invaded by water, i.e. an oil producing layer that has been partially invaded by water from a lower layer.

The object is to treat with acid solely the portion of the oil-saturated layer, to reduce the extent of deterioration therein, while the acid will have a natural tendency to invade the oil (petroleum) zone and the water invaded zone in a practically identical manner.

The acidizing techniques, as well as the acids, mixtures of acids or acid fluids to be used are well known to a man of the art. Mention is made in particular of hydrofluoric acid HF and the mixtures of hydrofluoric acid and hydrochloric acid HF + HCl, or again fluoroboric acid and its derivatives, the latter serving in particular to reduce the damage caused by the clays.

The techniques of foam generating and injection are also well known.

According to the invention use can be made of any one of these techniques, generation on the surface, generation in situ, air, nitrogen, CO_2 or N_2/CO_2 foam, as well as surfactants or foaming agents, equally well known.

There is also known in the art a technique using an acid in foam (the acid being integrated in the foam, which is quite different from the present invention).

The principle of diversion by means of a foam is also known.

The process described consists in injecting, initially, a "preflush" of surfactant into the ZT and ZFP zones (stage 1), followed by a foam in these same ZT and ZFP zones (stage 2), followed by a treatment fluid, in particular a specific acid fluid, (stage 4) composed of a combination of a treatment agent, in particular a conventional acid or acid fluid for the diversion, and a certain proportion of surfactant.

These three operations work in association. However, only stages 2 and 4 are essential, stage 1 (preflush) being nonetheless extremely useful to ensure better penetration and better placing of the products injected in stages 2 and 4. A well "shut-in" stage 3 can be inserted, if required, between stage 2 and 4. An appropriate shut-in period would be in the order of 1/2 hour.

The value of such a shut-in is demonstrated below.

Although it is not intended to be restricted by any particular theory, it is pointed out that the surprising results obtained can be explained in part by the following mechanisms.

The foam, which contains a high proportion of air, has the effect (since we obtain a diversion effect from the acid fluid) of very substantially reducing the permeability of the zones treated.

It is probable that the air contained in the foam is responsible for this reduction in permeability, at least to a large extent.

As mentioned earlier, the acid fluid in stage 4 contains, according to the invention, a considerable proportion of surfactant. The presence of this surfactant permits real cooperation between the acid fluid and the foam, the overall result being that, contrary to all expectations, the foam is capable of very strong resistance to the injection of the acid fluid. It can thus play its part as a diversion agent for a long period of time.

Surfactant preflush stage 1, which is not essential, does, however, permit better penetration of the foam.

Indeed, as the rock is wetted by the preflush surfactant, the following foam will not tend to lose its own surfactant and thus will not "break down".

As mentioned above, a well shut-in stage 3 of approximately 1/2 hour is strongly recommended.

The reasons for the beneficial effect of this shut-in have not been explained. It is thought, however, that the shut-in has a greater effect upon the foam located in the zone to be treated (ZT) which, hypothetically, is the zone of least permeability. Far more marked destabilization of the foam is observed in this zone (ZT) than in the high permeability zone (ZFP).

Thus the more marked flow of the foam in the

ZT zone than in the ZFP zone is reflected by a decrease in the amount of air in the ZT zone that is far more marked than in the ZFP zone. It follows from this observation that permeability will increase a little in the ZFP zone (but not sufficiently to prevent diversion) but that permeability will, above all, increase very substantially in the ZT zone.

One surprising result is thus that of creating a highly efficient diversion by using a product that is, a priori, a "fragile", non-resistant one, that is to say a foam.

Figure 3 illustrates surfactant preflushing, which is recommended. Owing to the difference in permeability, the surfactant invades the ZT zone less deeply than the ZFP zone.

Figure 4 represents foam injection (stage 2). The foam also invades the ZT zone to a lesser extent than the ZFP zone (approximately 3 times less).

Figure 5 represents the injection of acid fluid according to the invention (chiefly composed of ordinary diluted acid and surfactant).

It was noted surprisingly that the foam (9) was endowed with quite exceptional mechanical resistance to the injection under pressure of this acid fluid (10).

This cooperation effect is thought to be due to the presence of surfactant in the fluid and in the foam, which creates a very strong counter-pressure effect in the foam barrier.

Owing to the differences in penetration of the foam into the ZT and ZFP zones, the foam in the ZFP zone will present far higher resistance than the foam in the ZT zone. If the acid fluid is pumped at a pressure higher than the foam resistance threshold in the ZFP zone, the acid fluid is capable of invading the ZT zone but will not invade the ZFP zone.

Diversion by "pressure effect" is thus obtained.

The shut-in recommended in stage 3 primarily destabilises the foam present in the ZT zone. The shut-in thus makes it possible to obtain a substantial drop in the resistance threshold of the foam selectively in the ZT zone. It thus makes the diversion more efficient and increases the security of the acid fluid pumping operation, since it increases the divergence between the two resistance thresholds.

Generally, the acid fluid is pumped at a low flow rate. Use can, of course, be made of mixtures of well known surfactants. According to the invention, one can naturally use an air, CO₂, nitrogen, etc. based foam, the production and characteristics of which are familiar to the man of the art.

The invention also applies to the treatment of several superposed layers. Figure 8 represents the acid treatment with diversion for three layers ZT1, ZT2 and ZT3 with : ZT1 permeability < ZT2 per-

meability < ZT3 permeability. The surfactant preflush is injected (in the preferred manner), followed by the foam. The profile represented in figure 8a (left-hand side) is obtained.

A shut-in (optional but altogether preferable), followed by treatment with the fluid (acid + surfactant) according to the invention lead to a diversion by zones ZT2 and ZT3, the acid invading only zone ZT1 (figure 8, right-hand side).

The operation is then repeated for the two successive lower layers, as schematically represented in figures 8b and 8c.

The invention also applies to the treatment of wells partially invaded by water ("water coning"). This is the well known phenomenon, apprehended by producers, in which the water of a lower layer gradually invades the oil producing zone in the vicinity of the perforations.

To demonstrate the efficiency of the invention, use was made of two 24 D (24 Darcy) permeability test cells. The first is filled with brine, and the second with crude oil. A surfactant preflush (3 X the pore volume) is injected. The flow rates are substantially identical in the two cells.

A foam of brine + nitrogen-containing surfactant is then injected. We observe the formation of a barrier of foam, with an increase in the injection pressure, in the brine cell only. The acid fluid + surfactant according to the invention are then injected, at a low flow rate. We observe efficient diversion of the treatment fluid towards the oil zone. These sequences are represented in figure 10.

As mentioned earlier, the cells substantially reproduce the basic data for an actual well in Louisiana.

Figure 9 represents the treatment that could be applied to such a well according to the invention. Figure 9a represents the position of the foam in the "water cone" (in the oil zone, the foam has deteriorated during the shut-in).

We can then inject the acid which is efficiently diverted (Figure 9b) by the foam.

Figure 11 has been drawn on the basis of experimental results.

Two cells of compacted sand (50 cm long) were mounted in parallel.

The two cells have homogenous permeability, one of 24 D and the other 6 D. No counter-pressure is applied to the output of the system. The tests were conducted without any oil being present in the system. (As indicated earlier, oil destabilizes the foam barrier).

Stage 1 : A solution of surfactant (3 X pore volume considering the portion of the matrix that will be invaded by the foam) is injected into the two matrices. The surfactant used is a sodium/formaldehyde polynaphthalene-sulphonate (

a condensation product) at a volume concentration of 1 %, at 20 ° C. The flow rate through each cell is within the ratio of permeabilities (see Figure 11).

Stage 2 : A brine containing the surfactant, together with nitrogen, is injected at a rate of approximately 1.2 l/h under normal conditions (1.2 standard volume of nitrogen for 1 volume of brine + surfactant). The surfactant concentration in the brine is the same as for the preflush (stage 1), i.e. 1 % at 20 ° C.

In this test, the foam was not produced before entering the cells, but the gas and the liquid were injected in a diphasic form. The foam is formed immediately in the first few millimeters of the sample.

A very sharp rise in injection pressure is observed. A foam barrier of approximately 3 cm of 0.4 grade is formed in the 8 D cell and a foam barrier of approximately 8 cm of 0.7 grade is formed in the 24 D cell. The grade of a foam is known to be the ratio of the gas volume to the total volume of the foam.

Stage 3 : Shut-in of the well.

The decompression destabilizes the foam primarily in the low permeability cell. As indicated above, the advantage of a shut-in is that diversion is accelerated.

Stage 4 : In this test, the "treatment" fluid used is in fact an inert fluid constituted by a brine and surfactant. It is highly preferable for the surfactant concentration to be identical with that of stage 1, or close thereto. Injection takes place at a slow rate. A remarkable diversion is observed. (In figure 11, numbers 1, 2, 3 designate the above stages).

If the foam contains a mixture of surfactants, the treatment fluid must contain at least one of these surfactants.

All the surfactants of the preflush, the foam and the fluid must be compatible, and preferably identical.

Those surfactants suitable for foams in an oil-producing environment are numerous and well known. Simple routine tests will enable a man of the art to select the surfactant or surfactants to use, alone or mixed with one another, to obtain fluids that are mutually compatible and a stable foam, as well as compatibility with oil (no emulsions).

One particularly advantageous feature of the invention is that of treating the subterranean zone in several stages. This is particularly to be recommended when the height of the zone to be treated exceeds 20-30 inches (50-80 cm). The rule applied is one stage for every 20-25 inches (50-70 cm) in height.

According to the invention, and within the framework of the injection stages described above, it has been discovered that the efficiency of the diversion was surprisingly increased in the different

successive zones on condition that the volume of foam injected be increased, from one zone to the next.

Below is an example of implementation of the invention, in a gas well.

Stages :

A. Preflush with NH_4Cl (3 % solution) + 0.2 % non-ionic surfactant.

B. Treatment :

a. 500 gallons (2000 l) of cleaning fluid + 0.1 % of corrosion inhibitor.

b. HCl (10 %) + methanol (20 %) + 0.4 % corrosion inhibitor + 1 % surfactant + 2 % iron chelating agent + 300 scf/bbl (standard cubic feet) $\Delta 9\text{m}^3/160 \text{ l}$

C. Mud acid + methanol + corrosion inhibitor + iron chelating agent + nitrogen.

d. 3 % NH_4Cl (1300 gallons ; approximately 5200 l) + 10 % methanol + 1.5 % surfactant + 0.5 % of clay stabilizer + 300 scf ($\Delta 9\text{m}^3$) of nitrogen per bbl (160 l).

e. ± 8 bbl ($\Delta 1300 \text{ l}$) of Mitchell grade 65 foam prepared from 2.8 bbl (approx. 450 l) of stage d) product.

f. 1300 gallons (approx. 5200 l) of 10 % HCl + 20 % methanol + 0.4 % corrosion inhibitor + 1.0 % surfactant + 2.0 % chelating agent + 300 scf N_2 (approx. 9m^3) per bbl ($\Delta 160 \text{ l}$).

g. Mud acid + methanol + inhibitor + surfactant + chelating agent + 300 scf N_2/bbl (approx. $9\text{m}^3/160 \text{ l}$)

h. 1300 gallons (5200 l) NH_4Cl + methanol 10 % + 1.5 % surfactant + 0.5 % clay stabilizer + 300 scf (9m^3) N_2 .

i. ± 10 bbl ($\Delta 1600 \text{ l}$) of grade 60 foam prepared from 3 bbl (approx. 470 l) of the stage h) product.

j. 10 % HCL + methanol 20 % inhibitor 0.4 % + 1.0 % surfactant + 2.0 % chelating agent + 300 scf N_2/bbl ($9\text{m}^3/160 \text{ l}$).

K. Mud acid + 20 % methanol + 0.8 % inhibitor + 1 % surfactant + 2 % chelating agent + 300 scf N_2/bbl (approx. $9\text{m}^3\text{N}_2/160 \text{ l}$).

l. $\text{NH}_4 \text{Cl}$ (1300 gallons, approx. 5200 l at 3 %) + 10 % methanol + 1.5 % surfactant + 0.5 % clay stabilizer + 300 scf N_2/bbl (approx. $9\text{m}^3\text{N}_2/160 \text{ l}$).

In this treatment example, it can be seen that diversion by the foam was carried out in two stages (e and i), with different volumes of foam.

Naturally, the process can extend to more than two stages.

The details (known additives, volumes and percentages, etc...) of the above stages are not limitative, as a man of the art can easily appreciate.

However, the principle consisting in increasing

the volume of foam from one stage to the next is essential.

In an oil or analogous well, the methanol can be dispensed with.

Claims

1. Process of treating a subterranean zone (or formation) crossed by an oil, gas, water, geothermal or analogous well, whereby the treatment fluid is diverted towards the zone to be treated that is of lower permeability than that of at least one of the adjacent zones, and whereby a foam is used as a diversion agent, characterized in that there are injected a foam formed with the help of an inert fluid, a gas and at least one surfactant, then a treatment fluid that contains at least one of the surfactants contained in the said foam, apart from the active fluid itself, and in that, in the case of treatment of a zone in several stages, the volume of foam injected in each stage is increased progressively.
2. Process according to claim 1, characterized in that it additionally comprises a preliminary stage of flushing ("preflush") by at least one surfactant compatible with that or those of the foam and of the treatment fluid.
3. Process according to claim 1 or 2, characterized in that it comprises, between the foam injection stage and the treatment stage, a well "shut-in" stage.
4. Process according to any one of claims 1 to 3, characterized in that the surfactant is a sodium/formaldehyde polynaphthalene-sulphonate condensation product.
5. Process according to any one of claims 1 to 4, characterized in that the treatment is carried out at a low rate of flow.
6. Process according to any one of claims 1 to 5, characterized in that the treatment fluid is a mixture of diluted acid and surfactant.
7. Fluid for the treatment of a subterranean formation or zone crossed by an oil, gas, water, geothermal or analogous well, characterized in that it consists of a mixture of acid diluted for oil treatment and of surfactant.
8. Application of the process according to any one of claims 1 to 6 for the diversion of treatment acid in the field of oil production, or the diversion of treatment acid in the field of oil

production in the case of a well partially invaded by water.

9. Application of the treatment fluid according to claim 7 for the diversion of treatment acid in the field of oil production, or the diversion of treatment acid in the field of oil production in the case of a well partially invaded by water.

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Fig-1

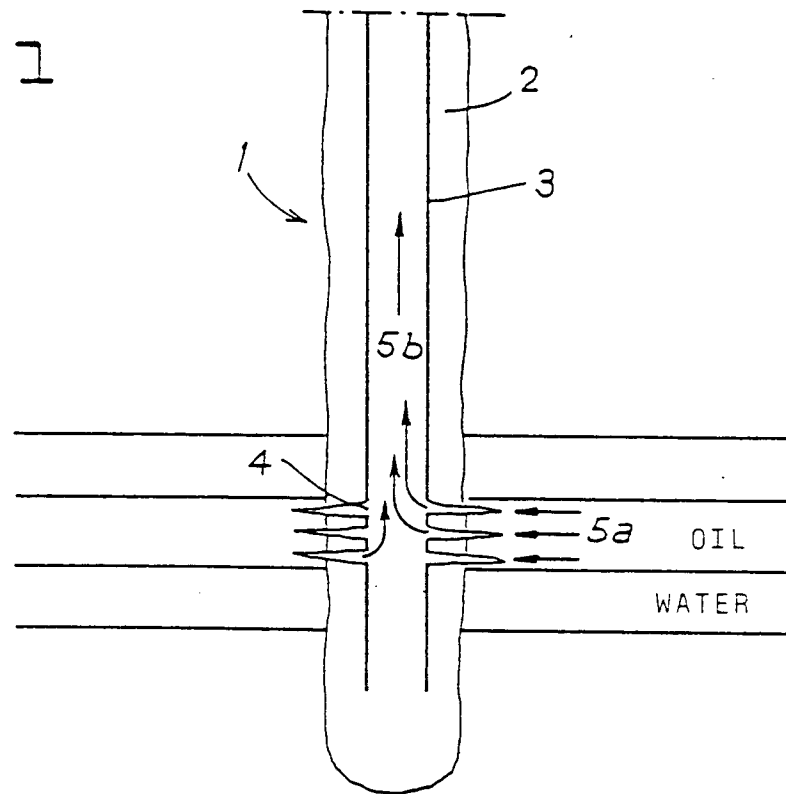


Fig-2

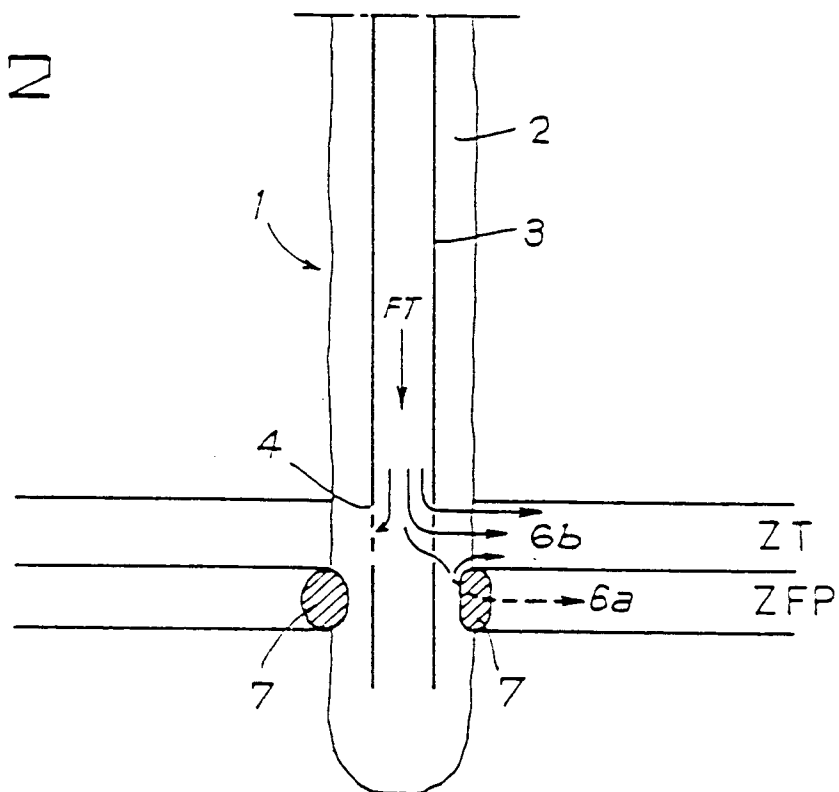


Fig. 3

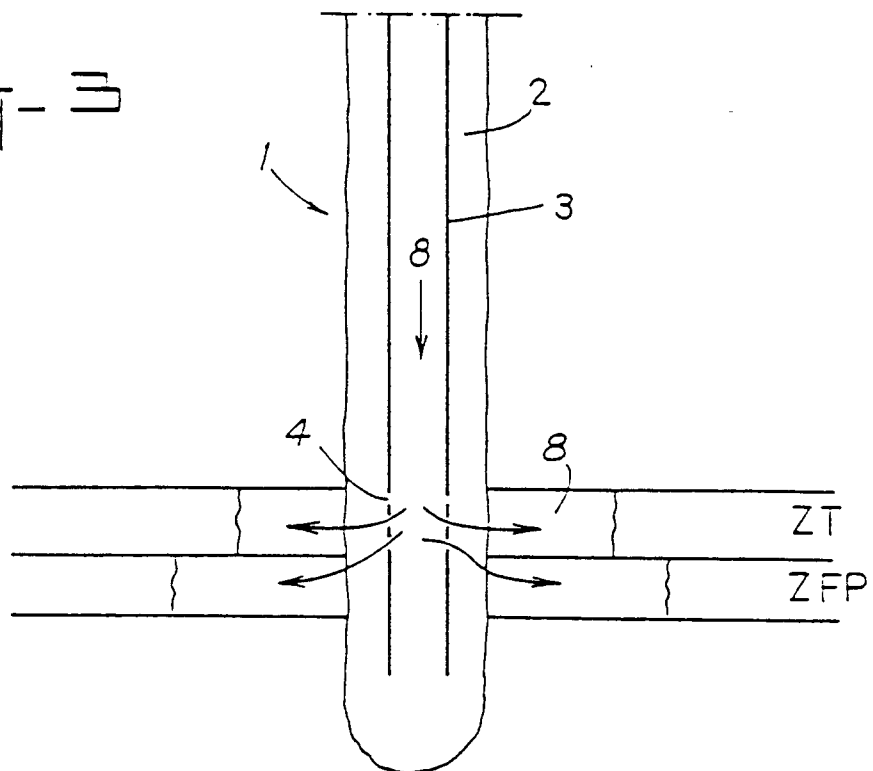


Fig. 4

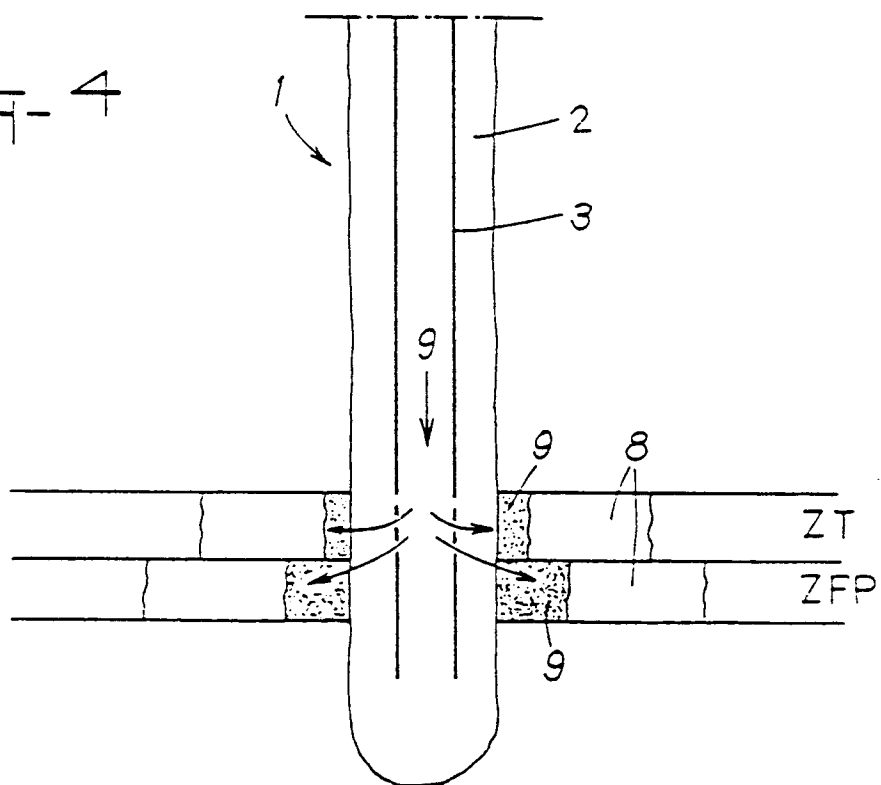
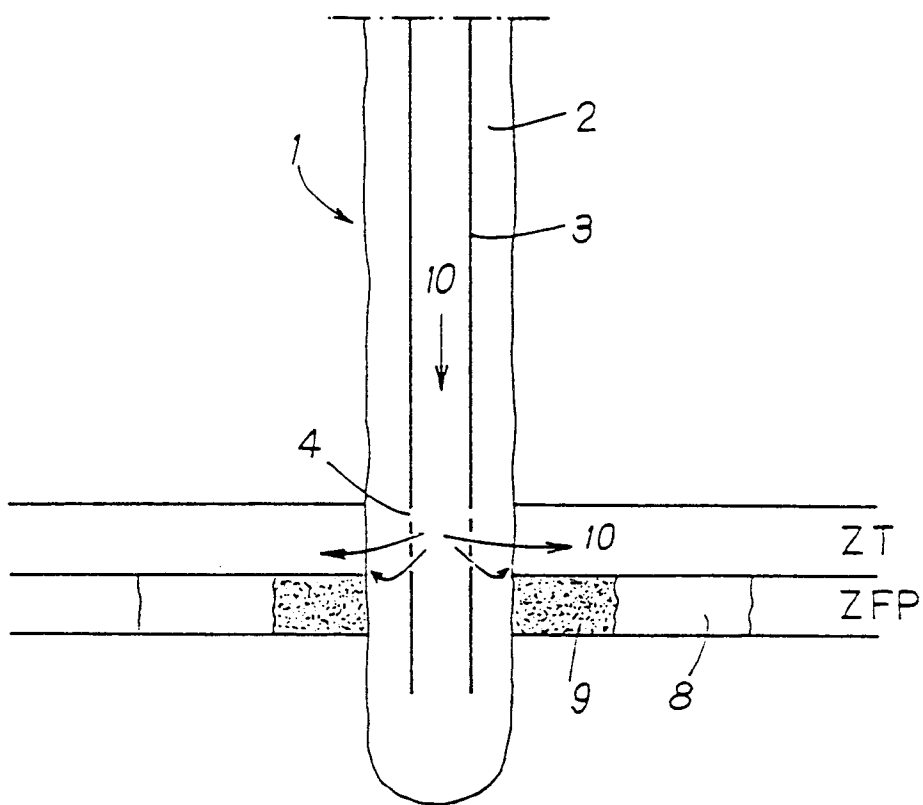
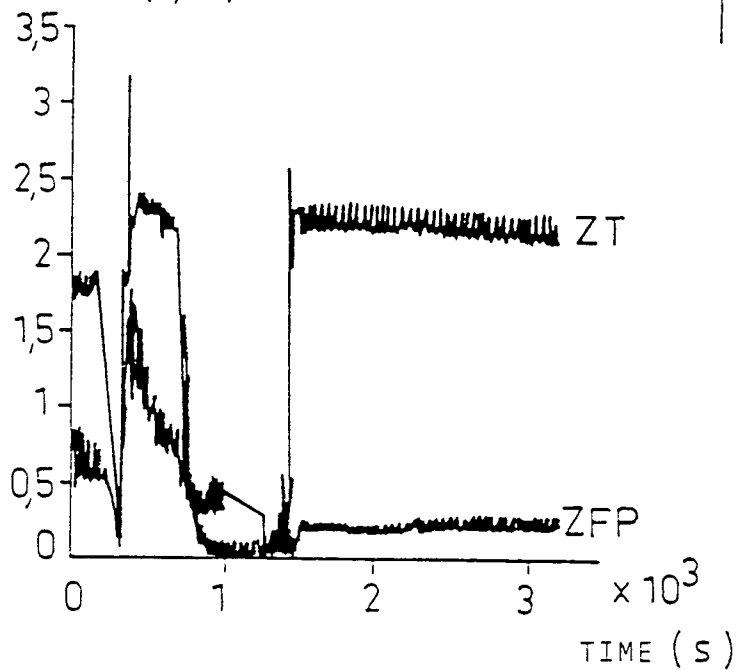


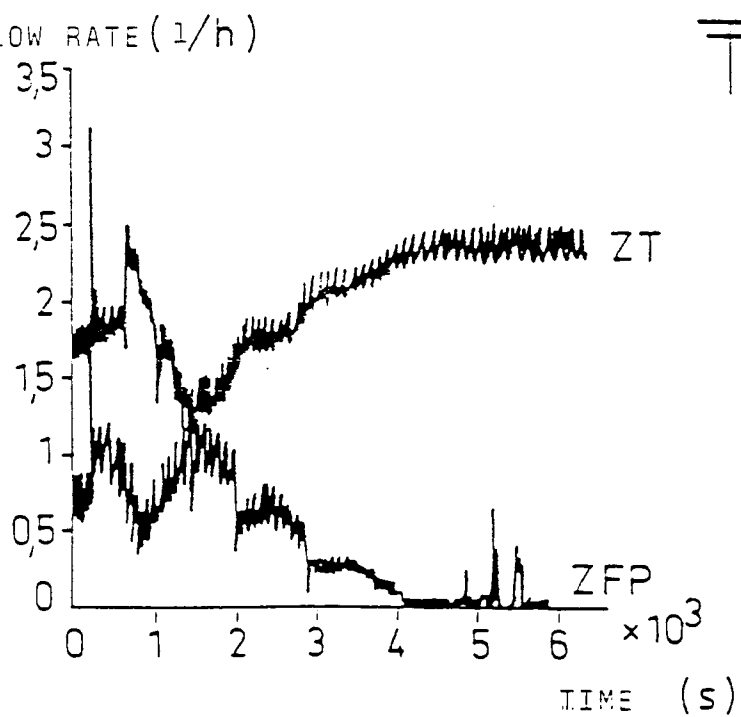
Fig. 5



FLOW RATE (l/h)

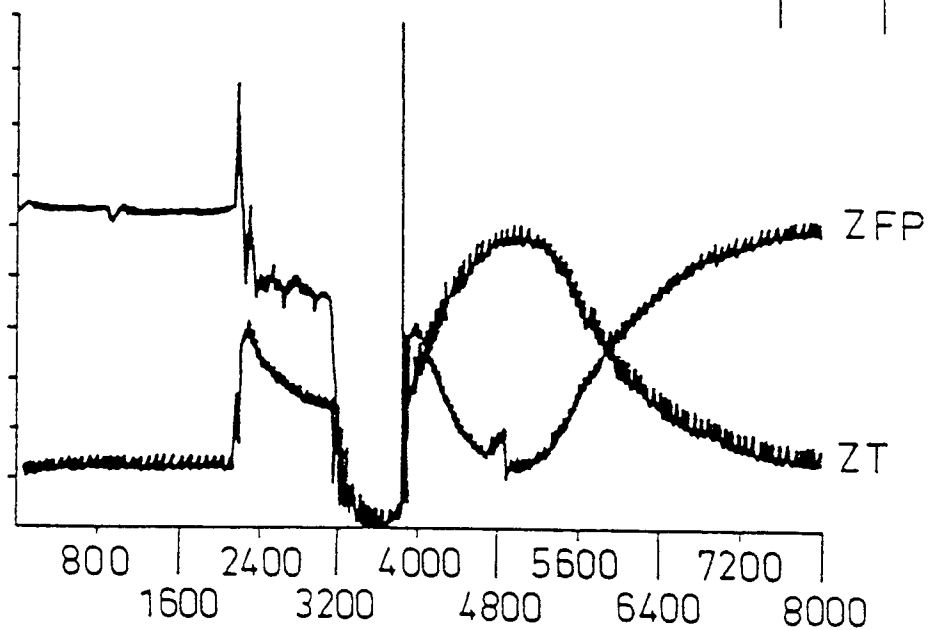


FLOW RATE (l/h)



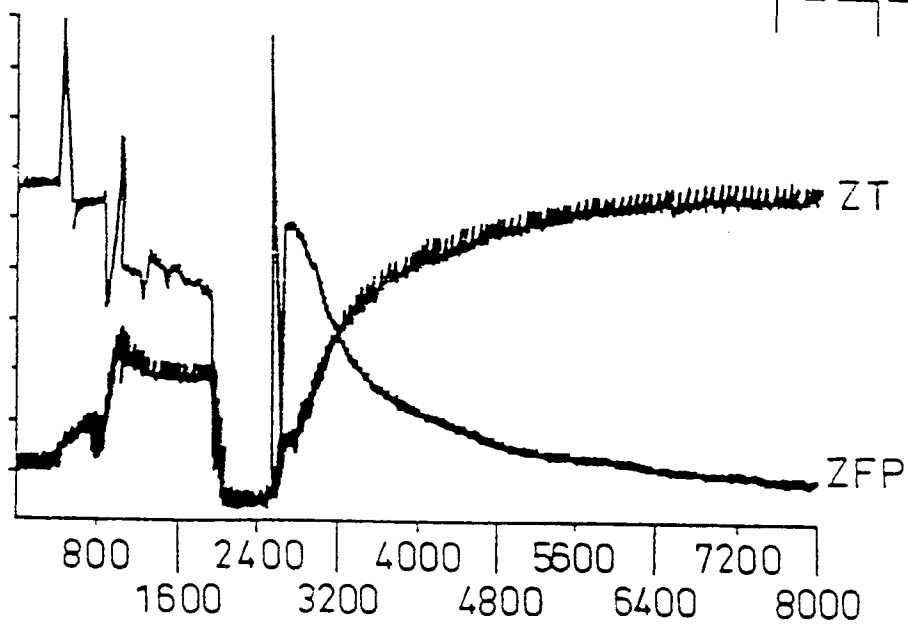
FLOW RATE (1/h)

T-9-7a



FLOW RATE (1/h)

T-9-7b



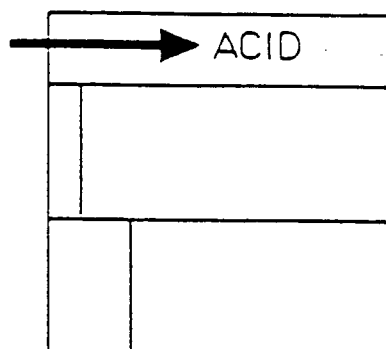
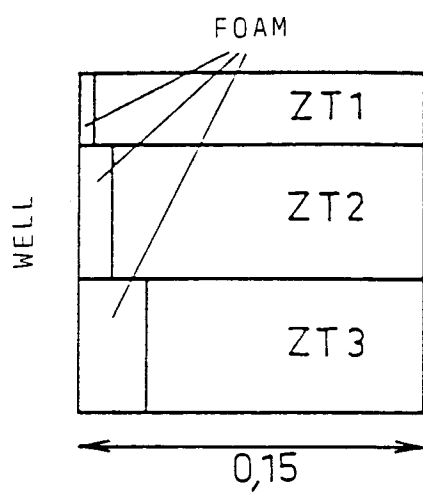


Fig. 2a

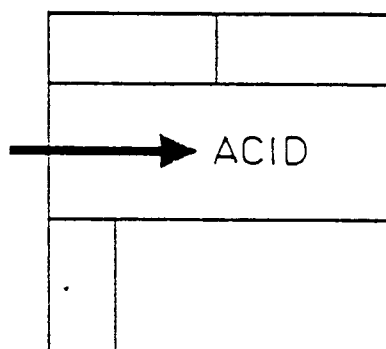
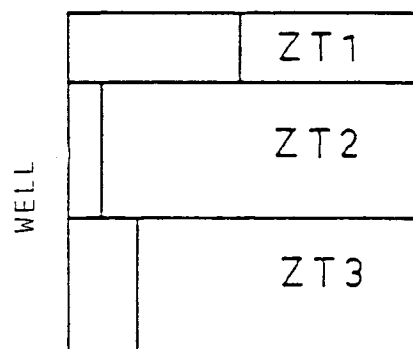


Fig. 2b

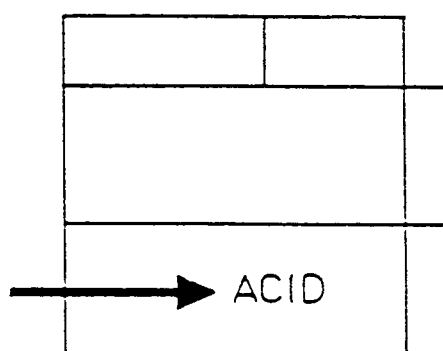
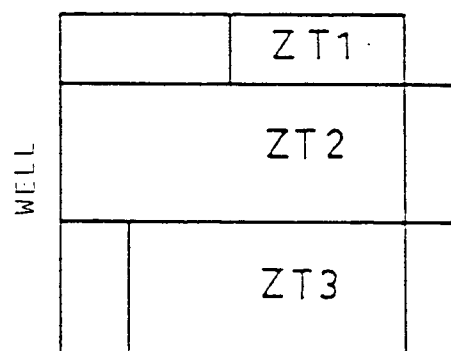


Fig. 2c

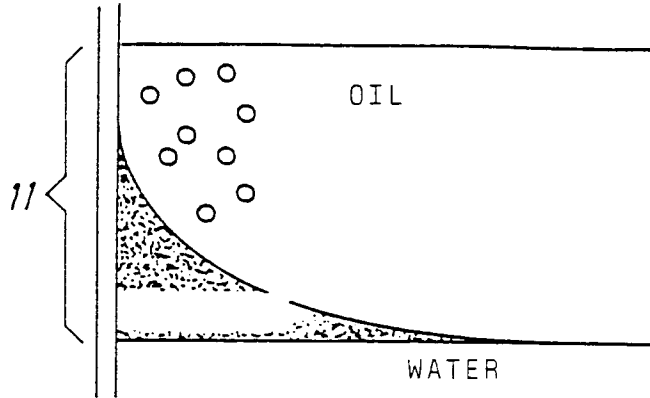


Fig. 3a

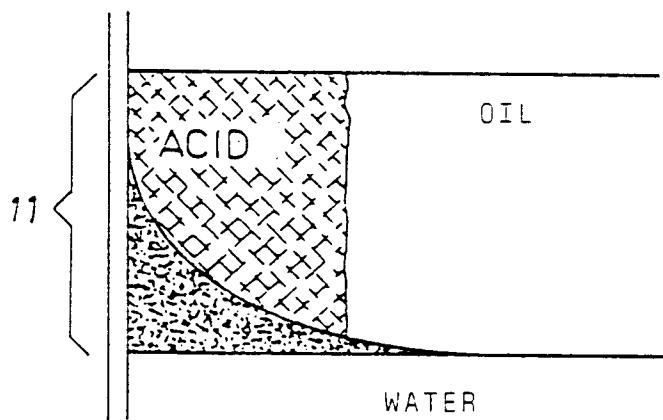


Fig. 3b

Fig-10

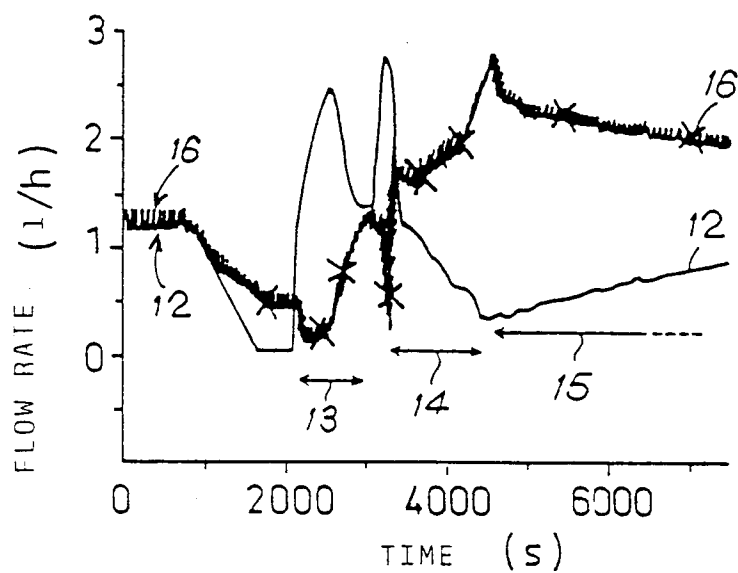
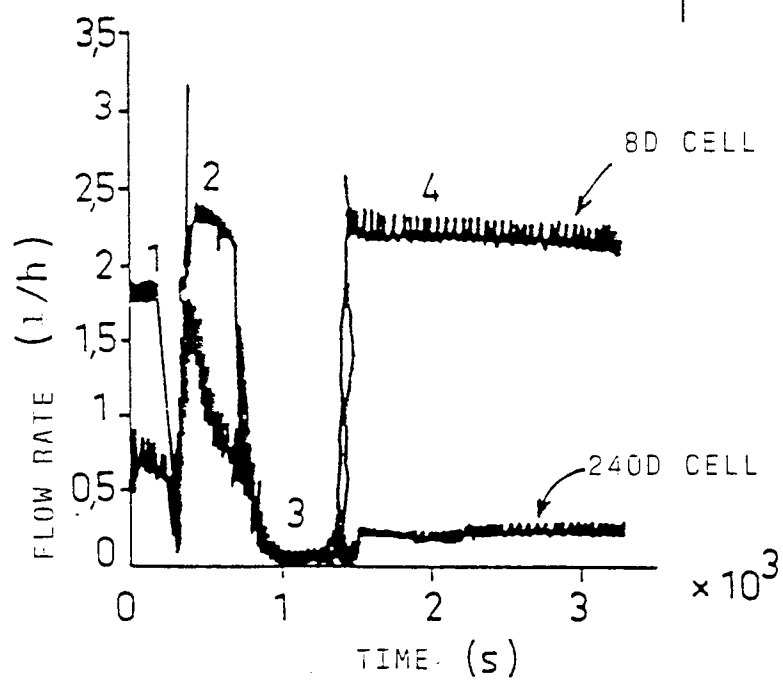


Fig-11





European Patent
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EUROPEAN SEARCH REPORT

Application Number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 91202222.5
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	EP - A - 0 305 612 (UNION OIL COMPANY OF CALIFORNIA) * Totality * --	1, 4	E 21 B 43/27
A	US - A - 3 612 179 (BYRON JACKSON INC.) * Totality * ----	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			E 21 B 43/00
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 02-12-1991	Examiner BENÇZE
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			